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Enzyme-Retted Flax Fiber and Recycled Polyethylene Composites

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Municipal solid wastes generated each year contain potentially useful and recyclable materials for composites. Simultaneously, interest is high for the use of natural fibers, such as flax (*Linum usitatissimum L.*), in composites thus providing cost and environmental benefits. To investigate the utility of these materials, composites containing flax fibers with recycled high density polyethylene (HDPE) were created and compared with similar products made with wood pulp, glass, and carbon fibers. Flax was either enzyme- or dew-retted to observe composite property differences between diverse levels of enzyme formulations and retting techniques. Coupling agents would strengthen binding between fibers and HDPE but in this study fibers were not modified in anyway to observe mechanical property differences between natural fiber composites. Composites with flax fibers from various retting methods, i.e., dew- vs. enzyme-retting, behaved differently; dew-retted fiber composites resulted in both lower strength and percent elongation. The lowest level of enzyme-retting and the most economical process produces composites that do not appear to differ from the highest level of enzyme-retting. Flax fibers improved the modulus of elasticity over wood pulp and HDPE alone and were less dense than glass or carbon fiber composites. Likely, differences in surface properties of the various flax fibers, while poorly defined and requiring further research, caused various interactions with the resin that influenced composite properties.

KEY WORDS: Flax fibers; enzymes; solid waste; milk containers; composite.

INTRODUCTION

Flax (*Linum usitatissimum L.*) is an agricultural crop grown on 12 million acres worldwide [1] that can provide food [2], fuel [3], and fiber [4]. Globally, the US is the largest per capita consumer of flax fiber and nearly all of this industrial grade fiber is imported. Studies conducted by DaimlerChrysler [5] indicate natural fiber automotive components require 83% less energy and are 40% less expensive than glass fiber components. Incorporating flax in thermoplastic materials provides reinforcement and could potentially lessen problems associated with the disposal of large quantities of municipal solid waste. In 2000, plastic constituted 10.7% of the generated 232 million tons of municipal solid waste, with 53.4 millions tons of this waste recycled [6].

Plastic waste (such as HDPE from milk containers) is currently recovered because it offers mechanical properties, recyclability, and potential material for various products. As a natural fiber, flax has a density of 1.5 g/cm^3, an elongation to break of 2.4%, a tensile strength of 1100 MPa and a Young's

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modulus of 100 GPa [7]. Flax fiber reinforces the biobased composite and reduces the amount of non-biodegradable materials. In 2002, 84% of the European composite market, containing wood and natural fiber, incorporated natural fiber, while in North America 10% consisted of natural fibers [8]. According to the US Department of Commerce, in 2001 the US imported over 18 million dollars worth of various grades of natural fiber flax. Industry analysts predict that demand for wood and natural fiber composites, and consequently raw natural fiber materials, will more than double between 2001 and 2006 [8].

Fiber and seed flax have been grown successfully across a large region of the US [1,9,10]. Compared to flax grown for seed, fiber flax plants are taller, have fewer branches, produce more fiber, have lower oilseed content and produce less seed [11]. Foulk *et al.* [12], Frederick [13], Loadholt [14], and Parks *et al.* [15] established that fiber flax could be grown as a winter crop in the south Atlantic region of the US with stalk yields around 6,700 kg per ha. Plant stalks can be dew-retted to separate fiber from non-fiber fractions, when attention is paid to produce uniform fibers that are not over—or under-retted [16]. A method is being developed to produce more uniform flax fibers by a process termed Spray-Enzyme-Retting (SER) [12,17]. Commercial pectinase-rich enzyme mixtures with calcium chelating agents applied to mechanically disrupted stems provide key steps in the development of a controlled and scientific approach to efficiently produce flax fibers of high and consistent quality. For uniform fiber quality, standards are being developed for industrial applications through the Flax and Linen Subcommittee, D13.17 of the Textile Committee of ASTM International.

Recycled HDPE is widely available, easy to process, and currently the most commonly used matrix material for wood composites [8]. For example, outside decking boards use HDPE as the matrix in 70% of its compounds. Physical properties of recycled HDPE milk bottles have shown little differences from virgin HDPE material [18]. Composites contain a medium such as HDPE and a high strength reinforcing agent. Natural fibers such as flax are sought in composites due to their strength, low abrasiveness, abundance, renewability, non-hazardous nature, biodegradability, low density, recyclability, low equipment requirements, and cost. The drawbacks of natural fibers include variable fiber quality, poor binding to matrix materials, chemical modification required for improved composite adhesion, and low processing temperatures.

Commercial pectinase-rich enzyme mixtures with calcium chelating agents produce flax fibers with specific properties [19]. A USDA Flax Fiber Pilot Plant, with a version of a commercial flax cleaning system (Unified Line, Czech Flax Manufacturing, Měřín, Czech Republic), is near completion at ARS–USDA, Clemson, South Carolina. At each point on this cleaning system, fiber and processing byproducts are produced with various properties for diverse industrial applications. Enzyme—and dew-retted flax stalks were processed through the pilot plant and then separated by passing through the Shirley Analyzer [19] into textile grade fibers and coarser textile byproduct fibers. The purpose of this study was to evaluate composites formed using the textile byproduct fiber from enzyme-retting via various formulations. Modifications via enzyme-retting could potentially impact binding and thus the stress transfer between the matrix and fiber.

MATERIALS AND METHODS

Materials

The source of materials included virgin HDPE, recycled HDPE, carbon fiber, glass fiber, thermal mechanical pulp (TMP), dew-retted flax fiber (cleaned on the Unified Line), and three levels of enzyme-retted flax fiber (cleaned on the Unified Line). Virgin HDPE (HDPE-5218) was supplied by Southeastern Polymers Inc., Greenville, SC. The recycled HDPE was obtained from milk jugs collected from the city recycle center in Clemson, SC. These milk jugs were cleaned and shredded for processing. Aircraft grade Thornel® carbon fiber (T-300, 309 NT grade with a fiber diameter of 7 μm) was supplied by Cytec Engineered Materials, Greenville, SC. Glass fiber (8 μm; Pyrex® fiberglass wool) was supplied by Corning Glass Works, Corning, NY. TMP was obtained from Bowater Inc. (Greenville, SC) a company that manufactures virgin newsprint grade TMP. Plastic composite substances such as outdoor decking typically use particulate wood flour while TMP is fibrous. Flax stalks were dew-retted in the field via indigenous microorganisms. These microorganisms introduce the natural color and property variations that are inherent with dew-retted flax fibers. Dew-retted flax stalks were processed on the Unified Line. This material was subsequently Shirley cleaned and only the coarser byproduct fiber was used for composite formation.

The enzyme-retted samples were 'Ariane' fiber flax shed-dried from South Carolina in 1999.

Production, harvesting, and yields of this winter crop have been provided in detail [16]. A recently developed enzyme-retting system [12,17] was used to separate the flax fibers from the epidermis/cuticle barrier and the lignified core (i.e., shive). Prior to enzyme-retting, dried flax stems were crimped through fluted rollers to disrupt inherent stem barriers (i.e., cuticle/parenchyma) and allow enzyme penetration into the tissues [19]. Flax fiber separation occurred using a series of enzyme (pectinase-rich) and chelator component formulations [19]. A commercial multi-enzyme product, Viscozyme L (Novozymes, Franklinton, NC), was combined in varying amounts with Mayoquest 200 (Callaway Chemical Co., Smyrna, GA) a commercial chelator product [19]. Enzyme and chelator levels studied respectively included the following: low (0.05% and 5 m*M*), middle (0.1% and 10 m*M*), and high (0.3% and 25 m*M*). Enzyme-retted flax was processed and cleaned on the Unified Line. This material was subsequently Shirley cleaned, and the coarser byproduct fiber was used for composite formation.

Preparation of Mixtures

All recycled HDPE/fiber composites were mixed to contain 30% fiber by weight. The controls were formed using only virgin HDPE or recycled HDPE. The quantity of material tested and testing conditions was consistent throughout all treatments. Mixtures were blended using a C.W. Brabender with computerized Plasti-Corder PL2000, which operates at a constant torque and speed using two counter-rotating intermeshing screws. The shredded pieces of recycled HDPE were first introduced into the C.W. Brabender PL2000 thermal mixer head for three minutes. This head was preheated to 175°C and mixed at a speed of 60 RPM. The various types of fibers were subsequently added to the molten HDPE and mixed for 2 min. This mixture was removed from the mixing head and fiber clumps within the molten HDPE were redistributed. These premixed materials were then reintroduced for another 2 min to redistribute the fibers throughout the mixture. This discontinuous process with a short mixing time of 4 min was performed to better maintain the length of flax fibers in the matrix for more effective fiber reinforcement.

Compression Molding

The plastic mixture was transferred to a compression mold to form Type V dog bone tensile test samples [20]. These dog bones were formed using a Dake hand-operated laboratory press (Model 44-250) in a compression mold at 175°C with a pressure of 4.14 MPa for 10 min. The mold was water-cooled to 60°C and the pressure removed. The dog bone tensile test samples were consequently obtained for testing.

Experimental Analysis

A minimum of six samples were prepared for each treatment. A factorial experimental analysis was performed to evaluate the mechanical and water absorption properties of the molded composites. The percent of fiber was constant throughout testing with duplicates of enzyme-retted flax at various enzyme and chelator levels. Specifically, the mechanical and physical properties determined were tensile strength, % elongation at break, modulus of elasticity, toughness, water absorption, swelling, and density.

Mechanical Testing

The tensile strength and modulus of elasticity for composites were determined by using the Instron testing instrument model 4202 [20]. The speed of testing was 2 mm/min. All test specimens were performed under the same conditions.

Density and Water Absorption

The density of the materials was determined by weighing and measuring the specimen's dimensions to the nearest 0.01 mm [21]. Water absorption and swelling of composites were evaluated by immersing 25.40 mm square samples in boiling water for 2 h according to a modified ASTM standard [22] with the weight and thickness of composites measured before and after the boiling water treatment. Sample thickness varied from 1.80 to 2.76 mm.

Fiber Length

Lengths of fiber within HDPE matrix were measured in a thin composite film using a Leitz Wetzlar Laborlux S microscope at 100X magnification. A Sony DXC-3900 3 CCD color video camera was used to capture the digital images. For each composite material, these digital images were manually analyzed and measured for fiber length using ImagePro Plus version 4.5.0.29.

RESULTS AND DISCUSSION

Recycled HDPE reveals impurities by a visual color difference from pure white virgin HDPE. The addition of carbon fibers results in a dark black composite. Visually the exterior of the glass fiber composite has an appearance comparable to recycled HDPE. TMP and the enzyme-retted flax fiber composites have a brownish tint. Dew-retted flax fiber produces a dark brown composite. Through microscopy, the evenly dispersed fibers were examined through thin films of the composite. Mean fiber lengths varied from 0.21 mm for TMP fibers to 0.40 mm for low level enzyme-retting (Table I). As expected for flax, particles of shive were visible with fibers surrounded by the polymer matrix. Visually, there were more dark particles in the dew-retted flax composite than enzyme-retted flax composites.

More studies are needed to identify these particles, the composite discoloration, and the influence of the shive material on composite properties. These shive particles may introduce weak spots and cause early cracking. In terms of exterior appearance, as an alternative for wood, the use of enzyme-retted flax fibers as reinforcement is more attractive than dew-retted flax fibers. Man-made fibers were uniform in diameter and varied only in mean length from 0.24 mm for glass to 0.30 mm for carbon. Despite the fact that specific steps were taken to maintain flax fiber length, future natural fiber processing modifications may be required for the most advantageous length and physical properties.

Flax fibers have a lower strength, density, and cost than glass or carbon fibers. The density of a composite frequently determines its application and is strongly related to the material strength. Density is often used to calculate the specific mechanical properties of a material. The density values of fibers, HDPE, and composite materials are shown in Table I. As expected for the higher density materials, the addition of 30% glass or carbon fibers increased the composites density to 1.09 and 1.05 g/cm^3, respectively. TMP demonstrated the lowest composite density of about 0.87 g/cm^3 followed by dew-retted and enzyme-retted flax fiber. The density of low enzyme-retted fibers (0.89 g/cm^3) was statically lower than dew-retted (0.94 g/cm^3), middle (0.96 g/cm^3), and high (0.97 g/cm^3) likely because of the larger amount of attached shive (woody portion) and cuticle (waxy) material.

Flax is cellulosic in nature and considered a hydrophilic fiber that absorbs water and results in composites with an undesirable dimensional change

Table I. Fiber, Polymer, and Composite Physical Properties[*]

	Fiber properties				Composite properties			
Sample	Strength (MPa)	Density (g/cm^3)	Diameter (μm)	Elongation (%)	Density (g/cm^3)	Fiber length (mm)	Water absorption (%)	Thickness swelling (%)
Virgin HDPE[a]	25.2[j]	0.96[j]	NA	9.5[j]	0.87 d	NA	0.011 d	0.75 c
Recycled HDPE[b]	25.2[j]	0.96[j]	NA	9.5[j]	0.90 d	NA	0.067 d	0.47 c
TMP[c]	1040[k]	0.36[k]	21[k]	3.7[k]	0.87 d	0.21 d	2.78 a,b	3.80 b,c
Dew-retted[d]	473.1 a	1.5[l]	32	1.67 a	0.94 c	0.32 b	1.57 c	5.03 a,b,c
Low[e]	213.9 b	1.5[l]	> 62	1.00 b	0.89 d	0.40 a	3.14 a	7.48 a,b
Middle[f]	324.5 b	1.5[l]	> 62	1.17 b	0.96 c	0.25 c,d	1.62 c	4.35 a,b,c
High[g]	295.2 b	1.5[l]	> 62	0.92 b	0.97 c	0.26 b,c,d	1.95 b,c	8.74 a
Glass[h]	2000[l]	2.52[h]	8[h]	2.50[l]	1.09 a	0.24 c,d	0.13 d	2.23 c
Carbon[i]	3750[i]	1.76[i]	7[i]	1.40[i]	1.05 b	0.30 b,c	0.14 d	1.81 c

[*]Values followed by different letters within columns are significantly different, $P < 0.05$, according to Duncan's New Multiple Range Test.
[a]Virgin HDPE obtained from Southeastern Polymers Inc., Greenville, SC.
[b]Recycled HDPE obtained from milk jugs collected from the city recycle center in Clemson, SC.
[c]Thermal mechanical pulp (TMP) was obtained from Bowater Inc., Greenville, SC.
[d]Fiber flax dew-retted, Unified Line cleaned, and subsequently Shirley analyzed.
[e]Fiber flax enzyme-retted: enzyme and chelator level (0.05% and 5 m*M*), Unified Line cleaned, and subsequently Shirley analyzed.
[f]Fiber flax enzyme-retted: enzyme and chelator level (0.1% and 10 m*M*), Unified Line cleaned, and subsequently Shirley analyzed.
[g]Fiber flax enzyme-retted: enzyme and chelator level (0.3% and 25 m*M*), Unified Line cleaned, and subsequently Shirley analyzed.
[h]Pyrex® fiberglass wool supplied by Corning Glass Works, Corning, NY.
[i]Thornel® carbon fiber (T-300, 309 NT grade) supplied by Cytec Engineered Materials, Greenville, SC.
[j]Properties of virgin HDPE obtained via The Matweb.com Online Materials Database [27].
[k]Properties of individual southern pine fibers [28].
[l]Properties of glass fibers [7].

and a low water resistance. Mechanical properties of water soaked composites are expected to deteriorate as the materials swell and absorb additional moisture [23]. Composite swelling increased from 0.47% up to 8.74% with the addition of natural fibers. Consequently, water absorption of composites increased from 0.07% to 3.14% when the natural fibers were added. In contrast, glass and carbon fibers demonstrated limited swelling and water absorption due to their hydrophobic nature. Swelling and absorption of natural fiber composites were inconsistent due to the varying amounts of fiber exposed on the composite's exterior of some but not all samples. Special outside layer considerations would be needed to better protect these natural fiber composites if such samples were to be used where exposure to moisture was likely. Processing refinements to exclude or limit surface fibers or a secondary covering process would result in a composite better suited to water exposure.

Coupling agents would strengthen binding between fibers and HDPE, but in this study fibers were not modified in anyway in order to assess differences in mechanical properties between the natural fiber composites. Flax was either enzyme—or dew—retted to observe composite property differences between different levels of enzyme formulations and retting techniques. Recycled HDPE is a material with useful properties and Table II demonstrates its strength of 25.6 MPa is not significantly different than virgin HDPE at 25.7 MPa. In this study, dew-retted fibers demonstrated a strength of 473.1 MPa while enzyme-retted fibers ranged in strength from 213.9 to 324.5 MPa. The addition of these natural fibers to the recycled HDPE significantly reduces the composites strength from 20.2 to 23.4 MPa. It appears that the lowest level of enzyme-retting and most economical formulation provides the same composite strength as the highest level of enzyme-retting. Enzyme-retted fiber composites are significantly stronger than dew-retted and TMP fiber composites, which do not significantly differ. Glass and carbon fibers have fiber strengths from 2000 to 3750 MPa and only boost the composites strength between 28 and 30 MPa. Carbon fiber contributes the highest strength to recycled HDPE, followed by glass fiber, flax fiber, and TMP fiber. For flax, stress concentrations and composite fractures likely arise due to residual woody shive material and poor binding between fibers, shives, and polymer. Figure 1 furthermore demonstrates how fiber reinforcement affects the stress-strain curve for various composite materials.

The inferior composite strength of natural fibers / HDPE is due to deficient bonds created between hydrophobic HDPE and hydrophilic fibers. However, the addition of enzyme-retted flax in HDPE composites nearly maintains the same strength as the recycled HDPE. Enzyme-retting is designed to produce uniform fibers with consistent properties. Prior to composite formation, dew-retted flax fiber strength properties are significantly larger than enzyme-retted flax fibers. Following composite formation, reduced tensile strength properties of dew-retted flax and TMP appear to confirm inferior bonding between unmodified natural fibers and HDPE. Epidermis / cuticle components contain higher calcium levels, resist removal, and are often observed in low quality textile fibers, with these fibers often demonstrating increased levels of waxes [24]. Enzyme treatments have been shown to alter the surfaces of flax fibers by removal of specific chemical components, with resultant modifications in yarn properties [25]. Flax fiber separation using pectinase-rich enzyme mixtures and chelator component formulations (to remove calcium) appear to modify the surface of flax fibers,

Table II. Composite mechanical properties*

Sample[a]	Tensile strength (MPa)	Percent elongation (%)	Modulus of elasticity (MPa)	Toughness (MPa)
Virgin HDPE	25.74 b	10.13 a	917.8 e	2.98 b
Recycled HDPE	25.61 b	9.33 b	738.0 e	4.36 a
TMP	20.24 d	2.26 e	1774.0 d	0.33 e
Dew-retted	20.93 d	2.47 d,e	2078.6 c	0.41 d,e
Low	23.20 c	2.92 c	2159.4 c	0.60 d
Middle	23.37 c	2.95 c	2192.4 c	0.58 d
High	23.11 c	2.81 c,d	2220.7 c	0.54 d,e
Glass	28.14 a	2.93 c	2636.8 b	0.82 c
Carbon	29.52 a	1.13 f	3148.2 a	0.32 e

* Values followed by different letters within columns are significantly different, $P < 0.05$, according to Duncan's New Multiple Range Test.
[a] Table I details sample identification.

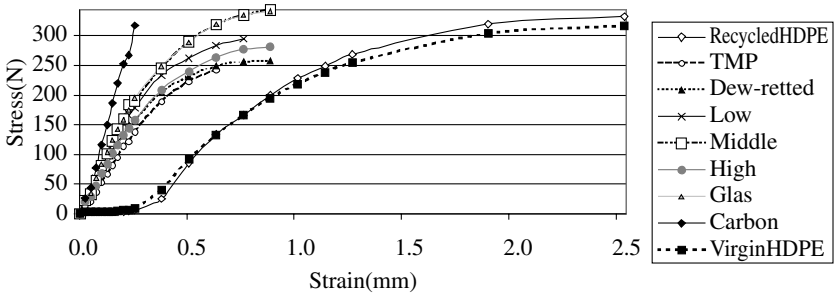

Fig. 1. Stress-strain curves for various composite materials.

remove pectin and calcium, and enhance bonding for composite formation. Better interfacial adhesion appears to occur with this modification of the fibers, resulting in better natural fiber matrix properties.

Modulus of elasticity is an important property because it represents the stiffness of the material and its resistance to elastic strain. Fibers high in crystallinity increase modulus of elasticity in composites [26]. Table II demonstrates that virgin HDPE and recycled HDPE have a similar ($P > 0.05$) modulus of elasticity. The addition of natural fibers in recycled HDPE increases the modulus of elasticity about 2–3 times from 1700 to 2200 MPa. The glass fiber and carbon fiber further increased the modulus of elasticity from 2600 to 3100 MPa, respectively. Statistically, there was no difference between the modulus of elasticity for flax composites (enzyme-retted vs. dew-retted) with samples approaching glass composite values. Flax fiber composites were formed with biodegradable and renewable resources thus producing materials that require a reduced amount of polymer with an increased modulus of elasticity. Additional in-depth evaluations of enzyme-retted fiber surfaces are required to better comprehend these results and for future fiber modifications. These values would likely increase with additional physical fiber modification, chemical modification, or coupling agents to promote binding. Further, maintaining the length of fiber flax could improve some properties.

Enzyme-retting performed at a pH of 5.0 may be a fiber modification process comparable to a traditional method of cellulose fiber modification termed mercerization using a high pH solution. Optimum conditions for enzyme-retting may further improve fiber separation in retting, improve composite tensile properties, and reduce water absorption. Mechanical properties of composites are dependent upon the strength and length of reinforcing fibers and how well the applied load is transmitted to these fibers [26]. These reinforcement properties are further dependent upon the quantity of fiber and a strong interface between the fiber and polymer. Processing modifications may be required to better maintain fiber length and improve composite properties.

Toughness is used to describe a combination of strength and ductility properties by evaluating the total area under the stress-strain curve (Fig. 1). Additional processing does affect polymers, and the virgin HDPE demonstrates a statistically higher elongation than recycled HDPE. Recycled HDPE has been somewhat degraded and results in a stiffened material that had poorer physical and chemical properties compared to virgin HDPE [18]. We found that recycled HDPE has a statistically higher toughness of 4.36 MPa compared to virgin HDPE whose toughness was 2.98 MPa (Table II). The addition of fibers substantially lowers the composites toughness to a range from 0.32 to 0.82 MPa. Glass fiber demonstrated the highest composite toughness of 0.82 MPa. Carbon fiber, TMP fiber, dew-retted fiber, and the high level of enzyme-retting fiber were statistically similar with the lowest toughness values.

CONCLUSION

The flax fiber/recycled HDPE composites were easily prepared through a thermal mixing process. This method, however, does not permit taking advantage of flax fiber lengths. Comparisons of the matrix materials (virgin HDPE vs. recycled HDPE) appear to demonstrate no detrimental effects upon the measured physical properties. Enzyme-retted and dew-retted flax fibers of various lengths and widths were randomly embedded with shives in the recycled HDPE matrix. Incorporation of flax fibers in a recycled HDPE composite varied the density, while increasing water absorption and swelling. Compared to recycled HDPE, physical properties of enzyme-retted composite materials demonstrated significant decreases in tensile strength, percent elongation,

energy absorption, and toughness while the modulus of elasticity was significantly larger. Composites with flax fibers from various retting methods, i.e., dew- vs. enzyme-retting, behaved differently; dew-retted fiber composites resulted in both lower strength and percent elongation. For the most part these enzyme-retted fiber composite materials were significantly stronger than TMP and dew-retted fiber composites. Statistically, there were no mechanical property differences between the highest enzyme-retting formulation and the most economical formulation. Various steps could strengthen binding between fibers and HDPE, but in this study fibers were not modified in any way in order to assess differences in mechanical properties between natural fiber composites. Further improvements would likely involve physical modifications, chemical modifications, or coupling agents to strengthen the interaction between hydrophilic shives and fibers and hydrophobic polymers. Flax fiber composites were formed with biodegradable and renewable resources, thus producing materials with reduced synthetic substances and an increased modulus of elasticity. Additional research is needed to understand and identify specific surface properties that occur with specific enzymes to tailor or improve composite characteristics.

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